DESIGN, CONSTRUCTION, AND OPERATION OF AN EXPERIMENTAL APPARATUS FOR CONTINUOUS COUNTERCURRENT SOLID-LIQUID EXTRACTION

A THESIS

Presented to

the Faculty of the Division of Graduate Studies

Georgia School of Technology

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Chemical Engineering

by

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June 1948

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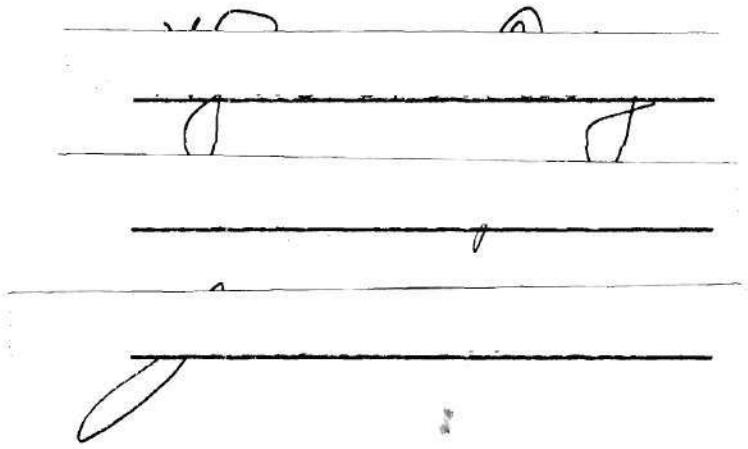
Approved:



Date Approved by Chairman May 18, 1948

ACKNOWLEDGMENTS

On the completion of this work I wish to express my sincerest thanks to Dr. J. W. Mason for his most valuable aid and guidance. I should also like to thank Dr. N. Sugarman for his assistance and Dr. W. M. Newton, who suggested the problem.

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INTRODUCTION

Solvent extraction has assumed considerable commercial importance within the past few years, particularly in the field of oil-bearing seeds. It offers greatest relative advantages in the case of seeds with low oil content. The oil content to which oil cake can be reduced by mechanical expression is approximately the same for all oil-bearing seeds, i.e. about five per cent. The yield of oil may be increased by as much as twenty per cent by substituting solvent extraction for mechanical expression.

Various types of continuous countercurrent extractors have been designed and are in use throughout the country. Most of them operate on the same general principle. The solvent is allowed to flow through a confined space countercurrent to the solid to be extracted. The solid is usually conveyed by a screw or in baskets pulled by an endless chain. Special types of equipment, such as that designed by Fauth[1] for peanuts, have been constructed for solids which disintegrate to a powder in extraction. A cost analysis of such processes in comparison with

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[1] P. L. Fauth, U.S. Pat. 2,253,696 (1941).

mechanical expression is found in a recent article by Bilbe.[2]

Among the other continuous extractors now in use is the Bollman[3] extractor, consisting of a number of baskets with perforated bottoms, which are carried by endless chains within a gas tight housing. Solvent is percolated through the baskets during their passage from the top to the bottom and back to the top again. The Kennedy[4] system has a series of rounded bottom tanks placed side by side in a gas tight compartment. Each tank has rotating within it a four-bladed paddle wheel with perforated paddles. By the scooping action of these blades the solid to be extracted is transferred from one tank to another countercurrent to the solvent.

Extraction rate data, which is essential for economic design of such equipment, is almost completely lacking. No point data on extraction batteries can be found.

The work of Boucher, Brier, and Osburn[5] has shed some light on mathematical data relative to diffusion. These workers made a study of both batch and countercurrent extraction using an artificially prepared solid of porous clay saturated with soybean oil. They found that the liquid film resistance was negligible as compared to the resistance to diffusion within the solid. Their continuous countercurrent extraction

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[2] C. W. Bilbe, Mechanical Engineering, 14: 10-14 (1937).

[3] Alton E. Bailey, Industrial Oil and Fat Products, 497-498 (1945).

[4] A. B. Kennedy, U.S. Pat. 1,628,787 (1927).

[5] D. R. Boucher, J. C. Brier, and J. O. Osburn, "Extraction of Oil from a Porous Solid", Transactions of the American Institute of Chemical Engineers, 38: 967-993 (1942).

apparatus consisted of a long tube through which the clay plates were conveyed in baskets passing countercurrent to the solvent. The point-to-point data obtained were found to be unreliable due to convection currents within the apparatus. However, it was noted that the experimental and calculated extraction times agreed reasonably well, which indicated that semi-batch extraction rate data could be used in predicting the extraction time for a continuous countercurrent system.

There are numerous methods of calculating the number of extractions required to produce a given oil concentration. However, the graphical methods of Ravenscroft[6] and Elgin[7] can be applied in a greater number of cases and give a clearer picture of the theory of extraction calculations.

The purpose of this work was to design and construct a suitable apparatus for obtaining rate data in a continuous countercurrent leaching system. Several runs have been made using the equipment to determine its approach to a true countercurrent system.

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[6] E. A. Ravenscroft, Industrial and Engineering Chemistry, 28: 851-855 (1936).

[7] J. C. Elgin, Transactions of the American Institute of Chemical Engineers, 32: 451-457 (1937).

APPARATUS

Due to the limited amount of space, the apparatus has been designed from the point of view of simplicity and ease of operation. The expense and space requirements for the usual mechanical type of extractor were prohibitive. Therefore, the equipment was designed on the principle of a diffusion battery.[8] This type of extractor consists of a number of individual units connected in series in such a way that units are added and removed from the system to simulate a solid flow. This is usually designated as a batch countercurrent system.

A battery of five extractors was made as shown in Figure 1. The glass tubes were made of fifty-seven millimeter pyrex glass of standard thickness. The ends of each tube were ground smooth enough to be fitted in brass plates with gas tight gaskets. A quarter inch brass plate was fixed permanently at the upper end of a two foot angle iron frame, and the tube was suspended by three-eighth inch tie rods between this plate and a second brass plate at the bottom. Each of the plates was fitted with a synthetic rubber gasket. An eighth inch stop cock was placed in the upper plate to permit exit of the air in the unit as it was filled with solvent. Details of the unit are shown in Figures 2, 3, and 4.

The extractors were connected in series with standard 1/4 inch brass piping in such a way that the solvent could flow from top to bottom of any of the units in series or parallel from left to right. A double union was installed at the bottom of each unit to provide a space

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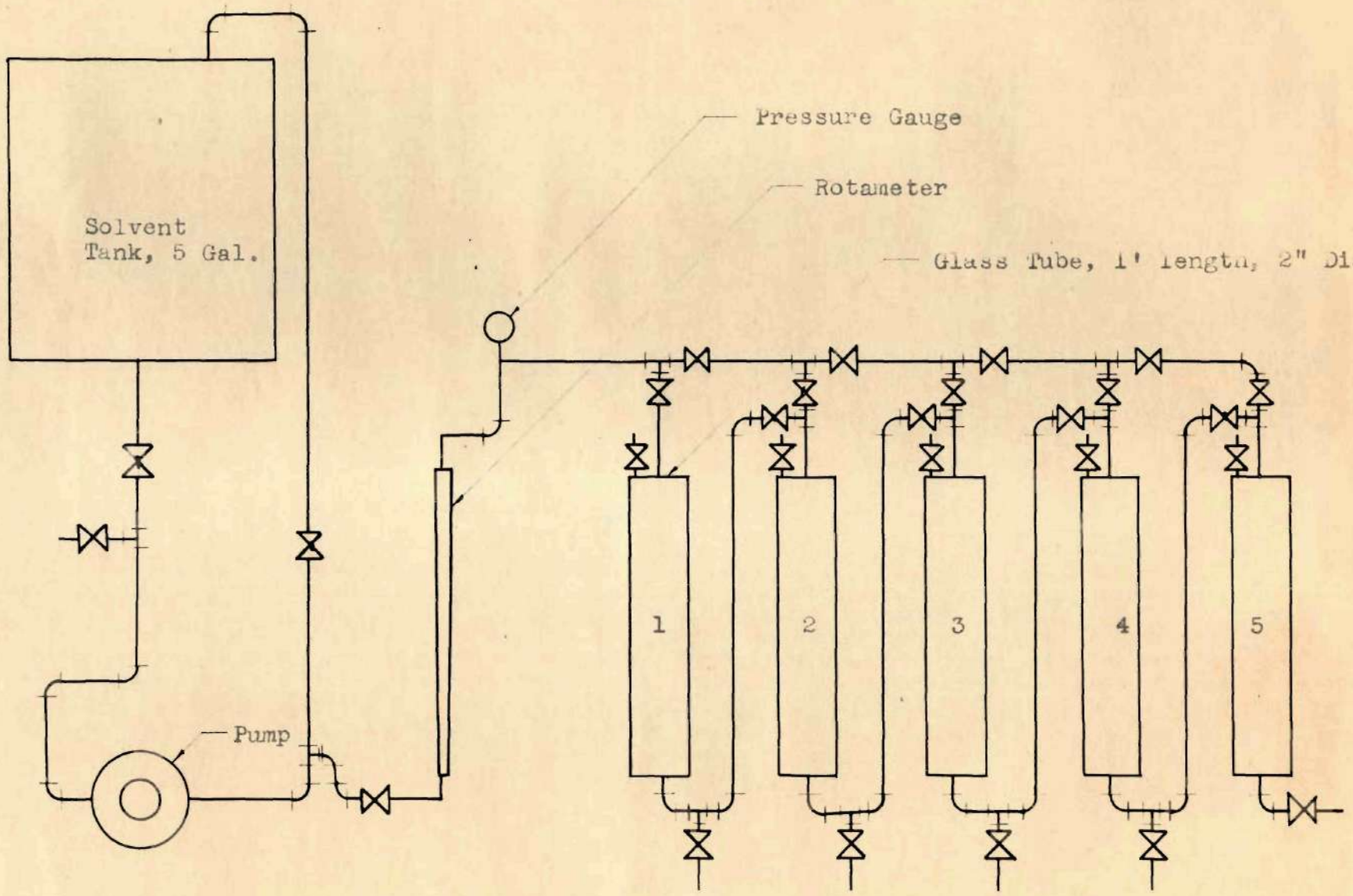
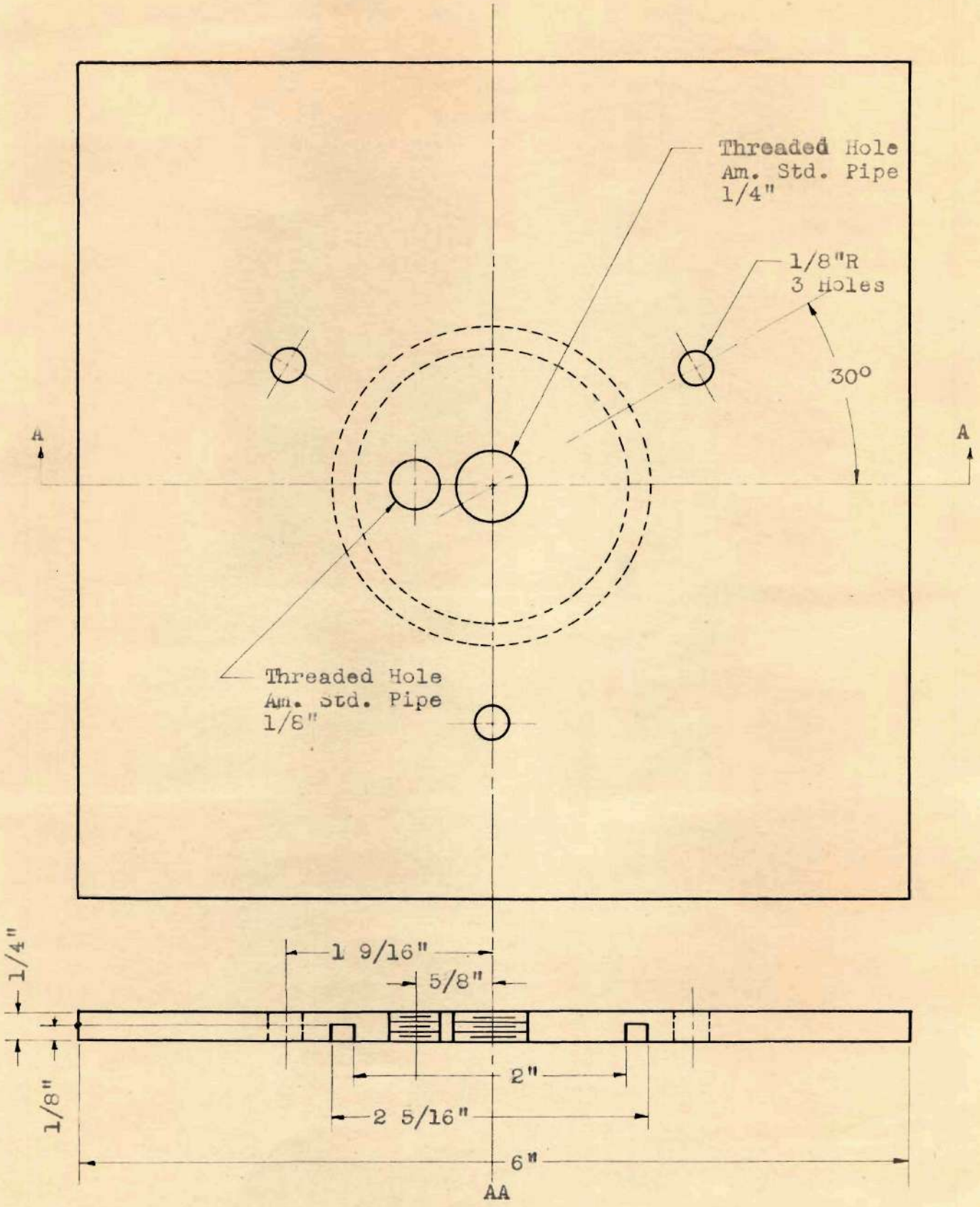
[8] Badger and McCabe, Elements of Chemical Engineering, p. 414 (1936).



Flow Sheet of Experimental Apparatus

Fig. 1.

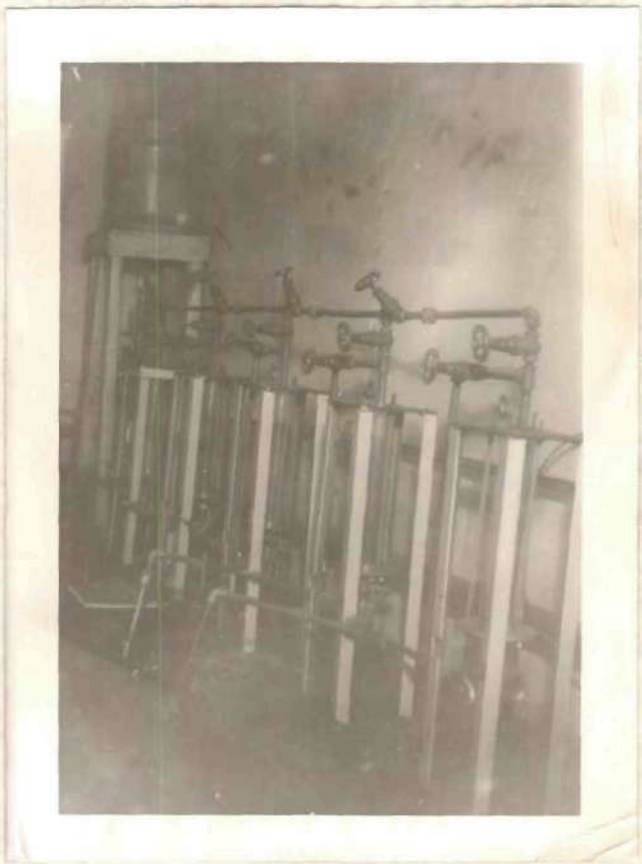


Details of Upper Brass Plate.

Fig. 2.

Two Units of the Apparatus

Figure 3 (Left)



Overall View of the Apparatus

Figure 4 (Right)

for lowering and removing the tube. Outlets were installed for removing sample solutions from each unit to obtain point data.

A rotameter, calibrated from zero to forty kilograms per hour of carbon tetrachloride, was placed in the system to measure the rate of solvent flow. A five gallon tank was connected through a 1/6 horse power gear pump to the rotameter with a by-pass line back to the tank. Globe valves were used for control of the solvent flow. A pressure gauge was placed in the line just past the rotameter as a safety device against high pressures and to indicate clogging within the lines.

One advantage that this type of apparatus might have over a single extraction tube is seen in the fact that there will be a great increase in turbulence which would help decrease the inaccuracy of point data due to convection currents.

DISCUSSION

Experimental Procedure

Runs were made to determine the accuracy of the apparatus for continuous countercurrent extraction and not specifically to get data on any particular system. Therefore, a solvent, carbon tetrachloride, was chosen which was not inflammable and not as volatile as commercially used solvents. Cottonseed flakes, as prepared for pressing, were selected as the solid to be extracted.

The flakes were first analyzed for total oil content by removing all oil in an eight hour Soxhlet Extraction with petroleum ether. The following results were obtained:

Cottonseed Flakes Analysis

Run Number	% Oil
1	36.28
2	36.32
3	36.31
Average	36.30

It was necessary to use some type of filter in the bottom of each tube to allow free flow of the solvent but at the same time prevent exit of the solid material. Various sizes of copper screen, soldered to the lower brass plate, were found to be too easily clogged. A layer of small glass balls was unsuccessful, since they provided too much resistance to the solvent flow. Next Berl Saddles were used unsuccessfully due to the solid particles escaping into the lines. The filtering system finally used consisted of a half inch layer of Berl Saddles intermixed with a small quantity of glass balls 1/8 inch in diameter.

In preparing for a three stage run, each of four units was first

prepared with the filter and then charged with two hundred grams of the flakes. Solvent was allowed to enter the stages until all were filled. The stop cocks were closed, and the run was ready to be started. The solvent was allowed to flow at a constant rate through three of the units for a definite period of time. This time was determined by the rate of solid flow desired. For example, if the solvent were allowed to flow for fifteen minutes, the equivalent solid flow would be eight hundred grams per hour. After that period, the first unit would be removed from the system and the fourth unit added to the system. If further time of extraction were desired, the second unit could be removed after a period of time and the fifth one added.

During the run, samples of the solutions were withdrawn every minute from the three units for analysis. The refractive index of each sample was determined using a Bausch and Lomb Precision Refractometer. From previously calibrated curves of refractometer reading versus concentration, the per cent by weight of oil was determined. The large spread of the refractive indices of the carbon tetrachloride and the oil, together with the sensitivity of the refractometer, made the analyses sufficiently accurate.

Samples of the solid leaving the extraction apparatus were analyzed by the Soxhlet Extraction in the same way as was the original cottonseed.

Discussion of Theoretical Calculations

For theoretical calculations, Elgin's[9] graphical method was chosen. A graphical method decreases the amount of calculation necessary, and, if expanded to a large enough scale, is just as accurate as any method. Elgin's method involves the use of triangular coordinate paper and can be enlarged to any scale with very little difficulty. It also possesses the advantages of generality, simplicity, ease of application, and the clarity with which the circumstances of a given situation may be visualized.

For an explanation of the theoretical calculations, it is necessary to derive the following simple material balances from Figure 5:

1. $W_n + L_0 = W_1 + L_3$
2. $W_nP_n + L_0S_0 = W_1P_1 + L_3S_3$ (Stages 1, 2, and 3)
3. $W_nP_n + L_1S_1 = W_2P_2 + L_3S_3$ (Stages 2 and 3)
4. $W_nP_n + L_2S_2 = W_3P_3 + L_3S_3$ (Stage 3)
5. $W_nP_n - L_3S_3 = W_1P_1 - L_0S_0$
6. $W_nP_n - L_3S_3 = W_2P_2 - L_1S_1$
7. $W_nP_n - L_3S_3 = W_3P_3 - L_2S_2$

The terms are defined in Table 1.

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[9] Elgin, op. cit.

Table 1

Definition of Symbols

L_0, L_1, L_2, L_3 : Weights per hour of solution leaving respective stages.

S_0, S_1, S_2, S_3 : Weight fraction of solute in solution leaving respective stages.

W_n, W_1, W_2, W_3 : Weights per hour of solids stream leaving respective stages.

P_n, P_1, P_2, P_3 : Composition of wet solids leaving respective stages expressed in weight fraction of oil remaining.

D_0, D_1, D_2, D_3 : Composition of dry solids leaving respective stages expressed in weight fraction of oil remaining.

The following assumptions must be made in order to use this method:

1. The system is composed of three components, solvent, solute, and carrier solid.

2. The carrier solid consists of a single substance of negligible solubility.

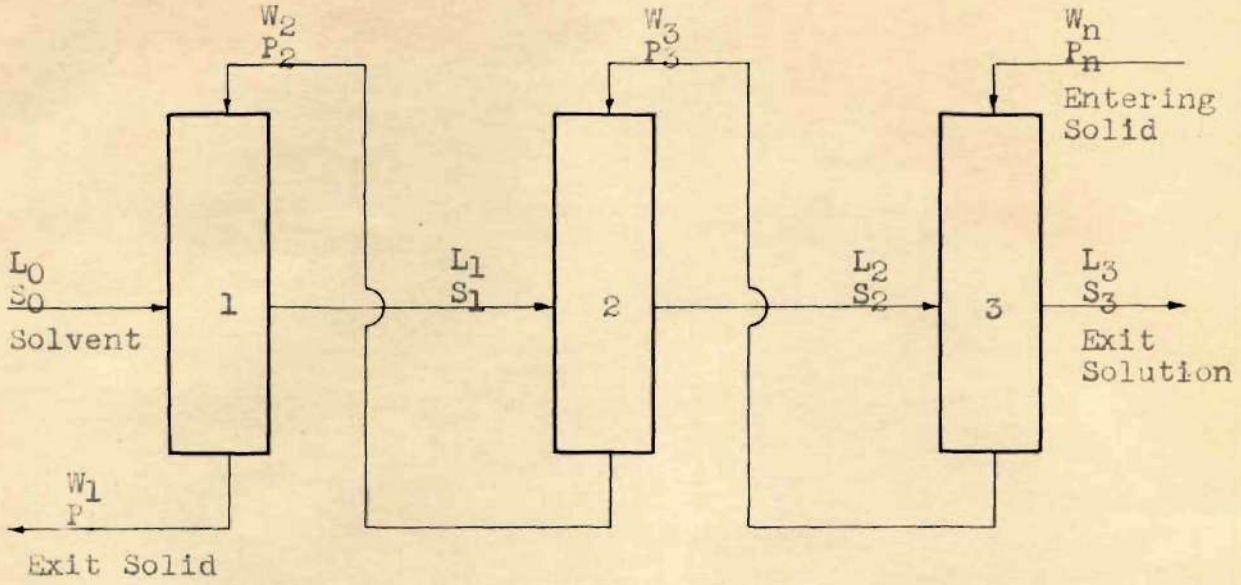
3. Only physical solution is involved.

4. The solute is not adsorbed by the carrier solid.

5. Equilibrium is attained in every stage; that is, the concentration of the solution retained by the solid is identical to that of the solution withdrawn.

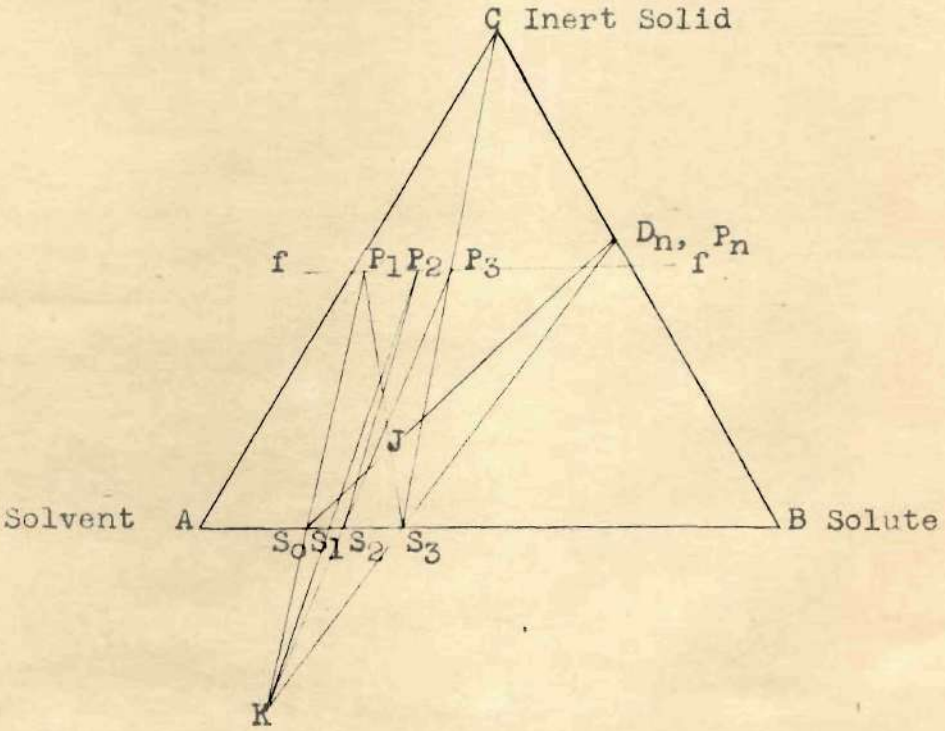
Figure 6 represents a simple triangular graph. From an understanding of this type of graph, it is seen that any mixture of the three components must lie within the triangle. Also, a third mixture formed by any two mixtures must be represented by a point along the line joining the two mixtures.

The line P_nS_0 is divided into the ratio of unit solution flow to



Theoretical Flow Sheet of Continuous Countercurrent Extraction.

Fig. 5.



Triangular Coordinate Diagram for Elgin's Calculations.

Fig. 6.

unit solids flow at the point J. Also, a line joining the exit solids composition (weight fraction of oil), P_1, to the exit solution composition, S_3, must pass through the point J. This is evident from equations 1 and 2.

Equations 2, 3, and 4 are simple material balances over the stages as indicated. The right sides of equations 5, 6, and 7, which are identical with 2, 3, and 4, may be considered as imaginary mixtures whose composition and quantity are produced by the subtraction of the oil in the solvent stream leaving any stage from the weight of oil in the solids stream entering that stage. These imaginary mixtures are all identical (as seen from the left sides of equations 5, 6, and 7), and will lie along a line joining the composition of the solids stream entering any stage to the composition of the solvent stream leaving the same stage. This is a point common to all such lines. It generally lies outside the triangle, such as at K.

The line ff divides the triangle into the ratio of the weight of solution retained by the solids to the weight of the completely drained and dried solids. This may or may not be a straight line and must be determined experimentally. The intersection of the line joining C and any composition along AB with ff is the point giving the composition of the wet exit solids. These lines are known as tie lines.

In conjunction with tie lines, the lines locating point K may be used for stepwise graphical calculations. By knowing the initial solvent composition, the location of the line ff, and the initial and final solids compositions, the concentration of oil leaving each extraction unit may be calculated.

To determine the proper location of the ff line, experimental data were collected by determining the weight of the wet solid from each extractor in a run. The wet solid was pressed to remove as much of the retained solution as possible, and then dried and weighed. The following data were obtained. The ratio given is that of the weight of solution retained by the solids within each stage to the weight of the completely drained and dried solids.

Stage	Gm. Wet Solid	Gm. Dry Solid	Gm. Solution	Ratio
1	325.7	158.0	167.7	1.06
2	322.1	153.6	168.5	1.08
3	313.4	150.7	162.7	1.08
4	320.5	164.6	155.9	.99

From this data, it was assumed that the ff ratio remained constant at 1.05, an average value. The only source of error involved here lies in the fact that a portion of the solution could not be pressed from the wet solids, and a certain quantity of the dissolved oil was left behind upon complete drying of the solids. Since the greatest concentration of oil in solution was less than seven per cent, the variation of the ff ratio could have been no more than .02 if as much as half the dissolved oil had been left in the dry solids. The error was considered negligible, since it was much less than the human error involved.

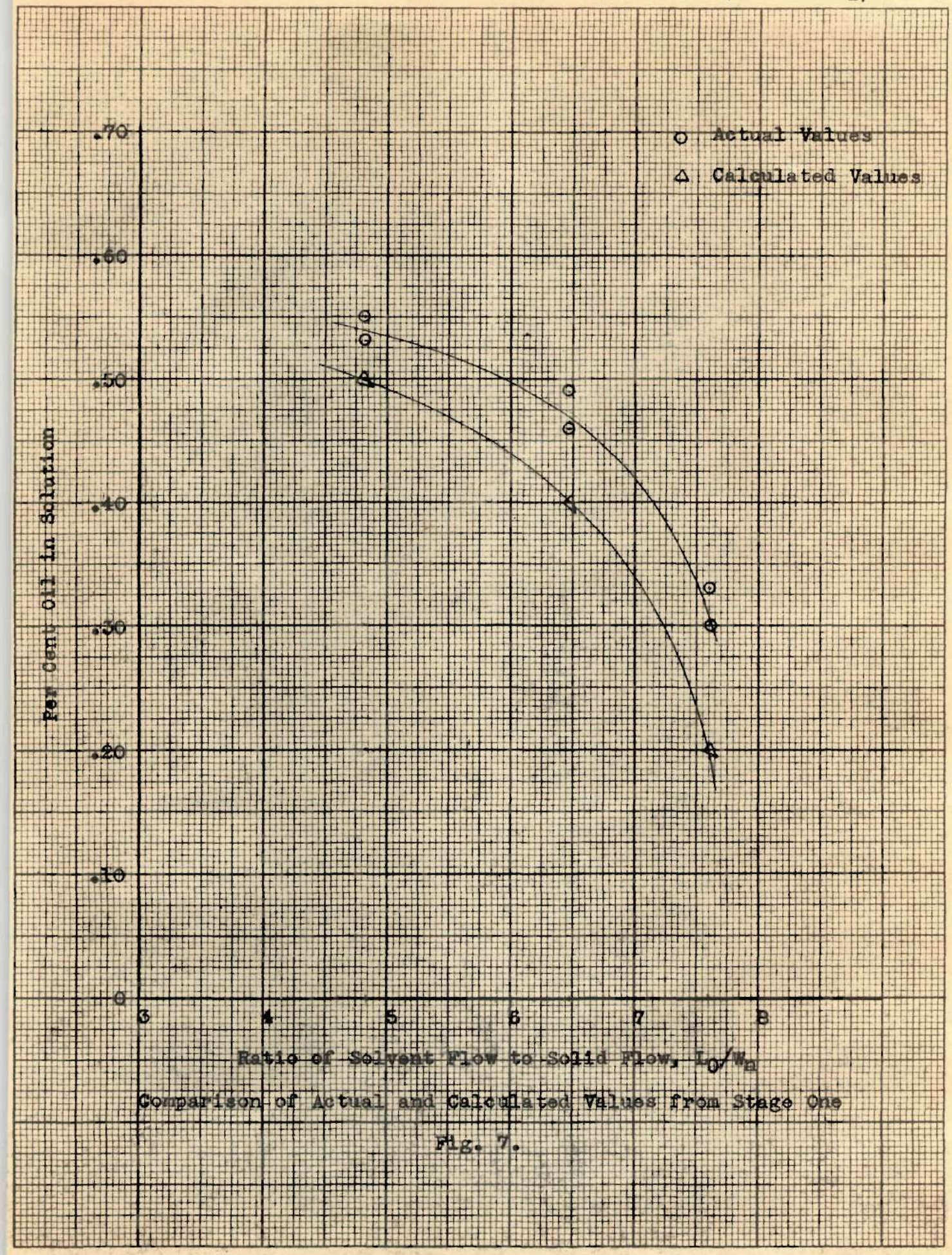
Since all the concentrations were less than seven per cent, the scale of the triangular paper was expanded to two and one half times its original size. Therefore, the regular size paper included in the calculations shows only forty per cent of the triangle; and K and the ff line are missing. The small paper was superimposed on an enlarged triangle and calculations were made.

Discussion of Results

The choice of cottonseed flakes as a solid was unfortunate in one way. The flakes had a tendency to disintegrate to a fine powder after several minutes of extraction. This not only clogged the lines during many runs, but also prevented a study of flake size from being made. It also added a possible source of error, since particle size has a large effect on extraction rate. However, it was noted that in runs with increased rates of solid flow, the disintegration did not have time to take place to any large extent.

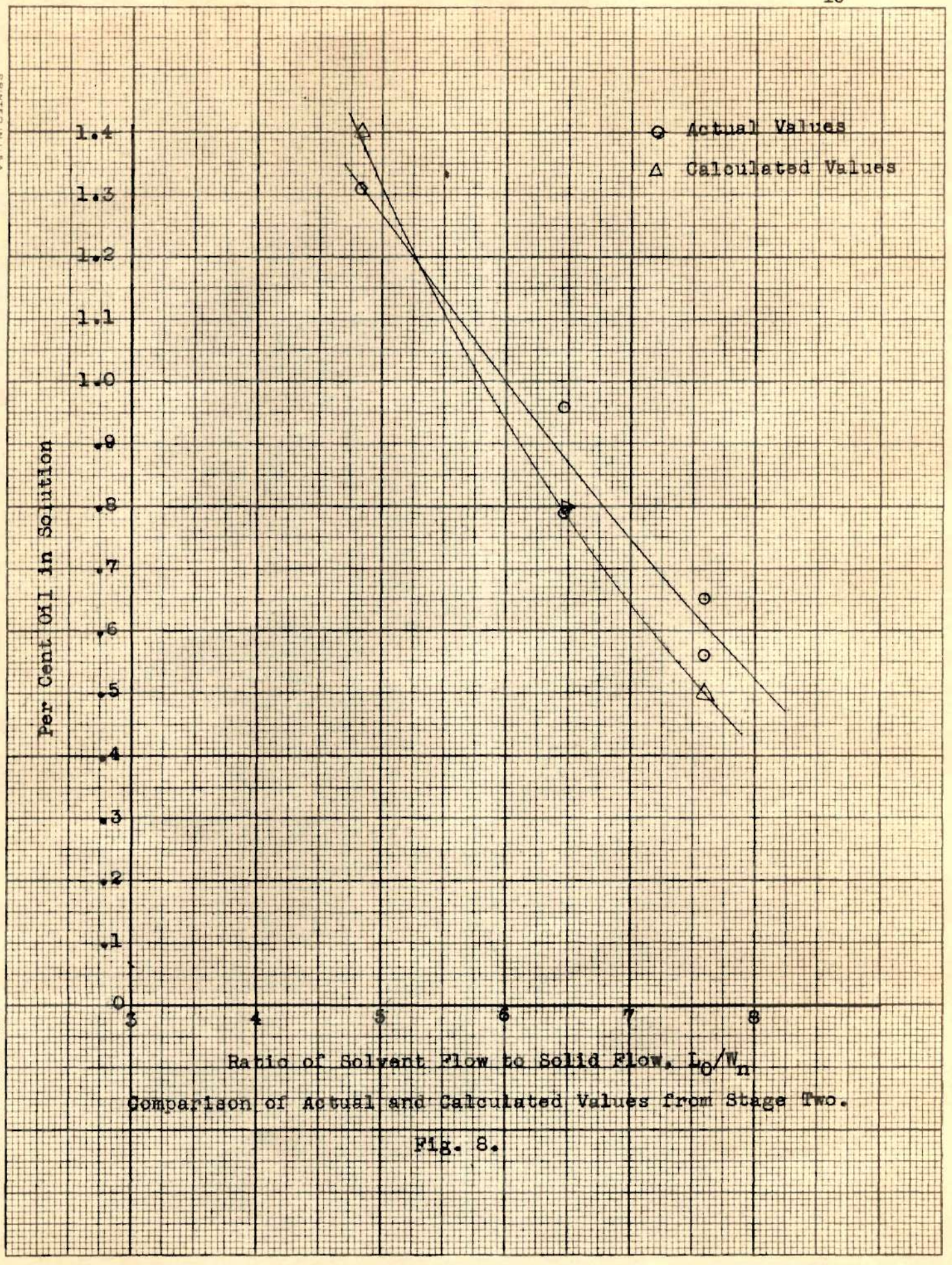
Figures 7, 8, and 9 show the comparison of actual and calculated results for the individual stages. The actual values are those obtained upon analysis of samples from the various stages during the runs. The calculated values are those obtained from Elgin's triangular coordinate method of theoretical calculations. The distance between the two curves shows the proximity of the actual values to true equilibrium conditions. The end point data compared much more favorably than did the point to point data. This is possibly due to convection currents within the system, which could be avoided by the use of a solvent and solute whose specific gravities are closer to one another.

Since only five stages were put in the extractor, the rate of solid flow was made considerably lower than desired. The time of each run had to be of a certain length to approach equilibrium conditions. This made it difficult to hold either solid or solvent rate of flow constant for a series of runs. Therefore, both were varied over smaller ranges; and their ratios were plotted for both actual and calculated values rather than their rates.



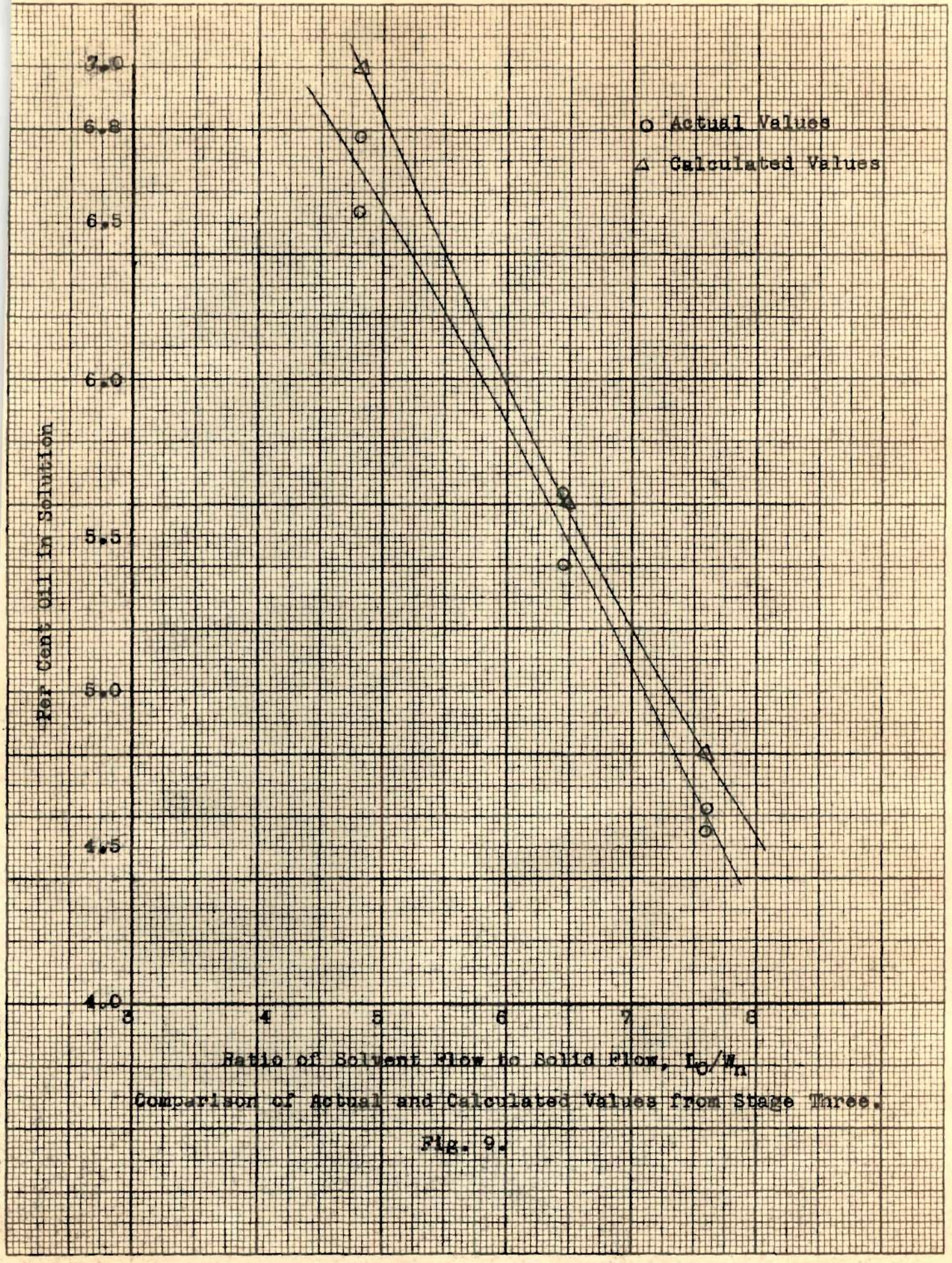
Comparison of Actual and Calculated Values from Stage One

Fig. 7.



Comparison of Actual and Calculated Values from Stage Two.

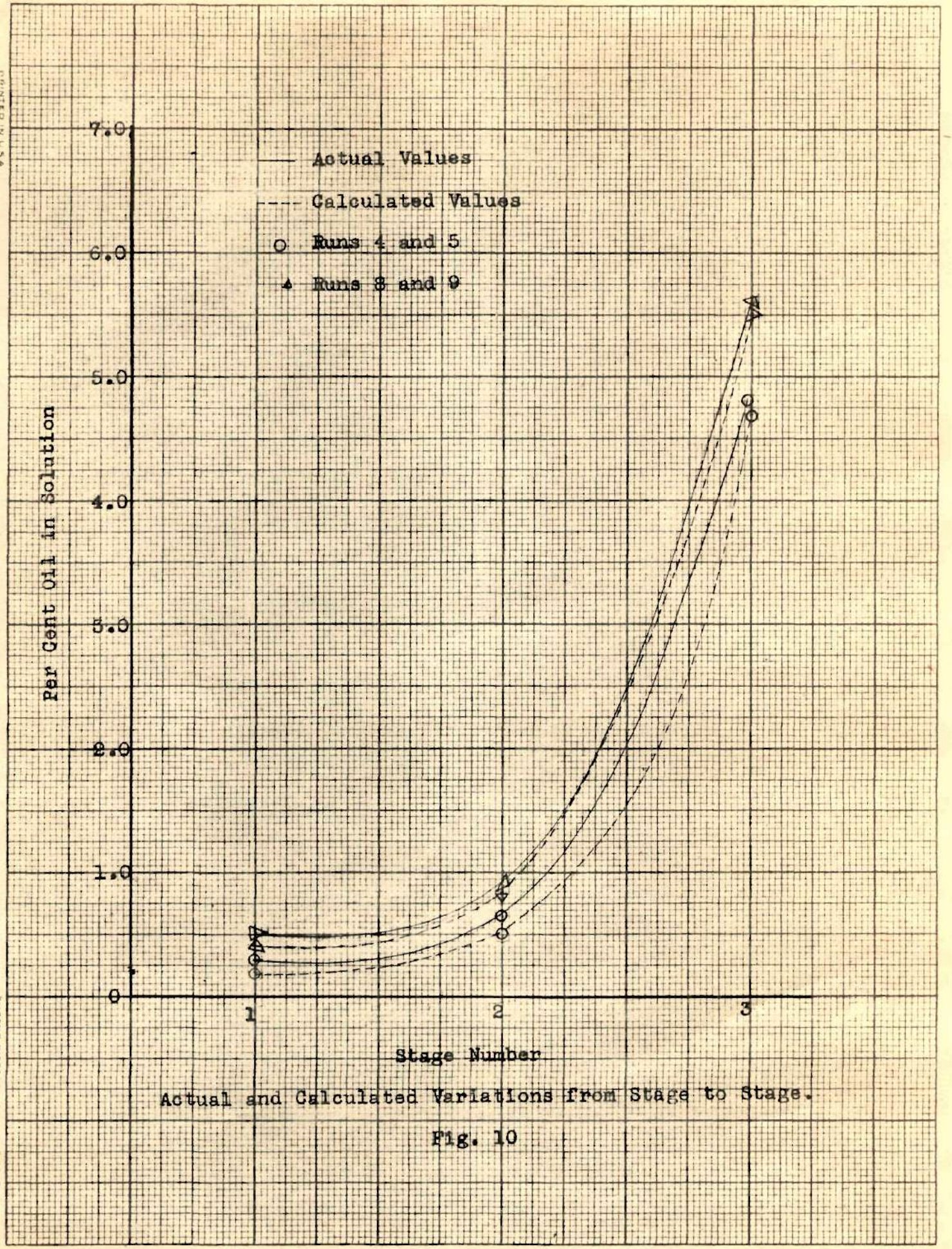
Fig. 8.



Comparison of Actual and Calculated Values from Stage Three.

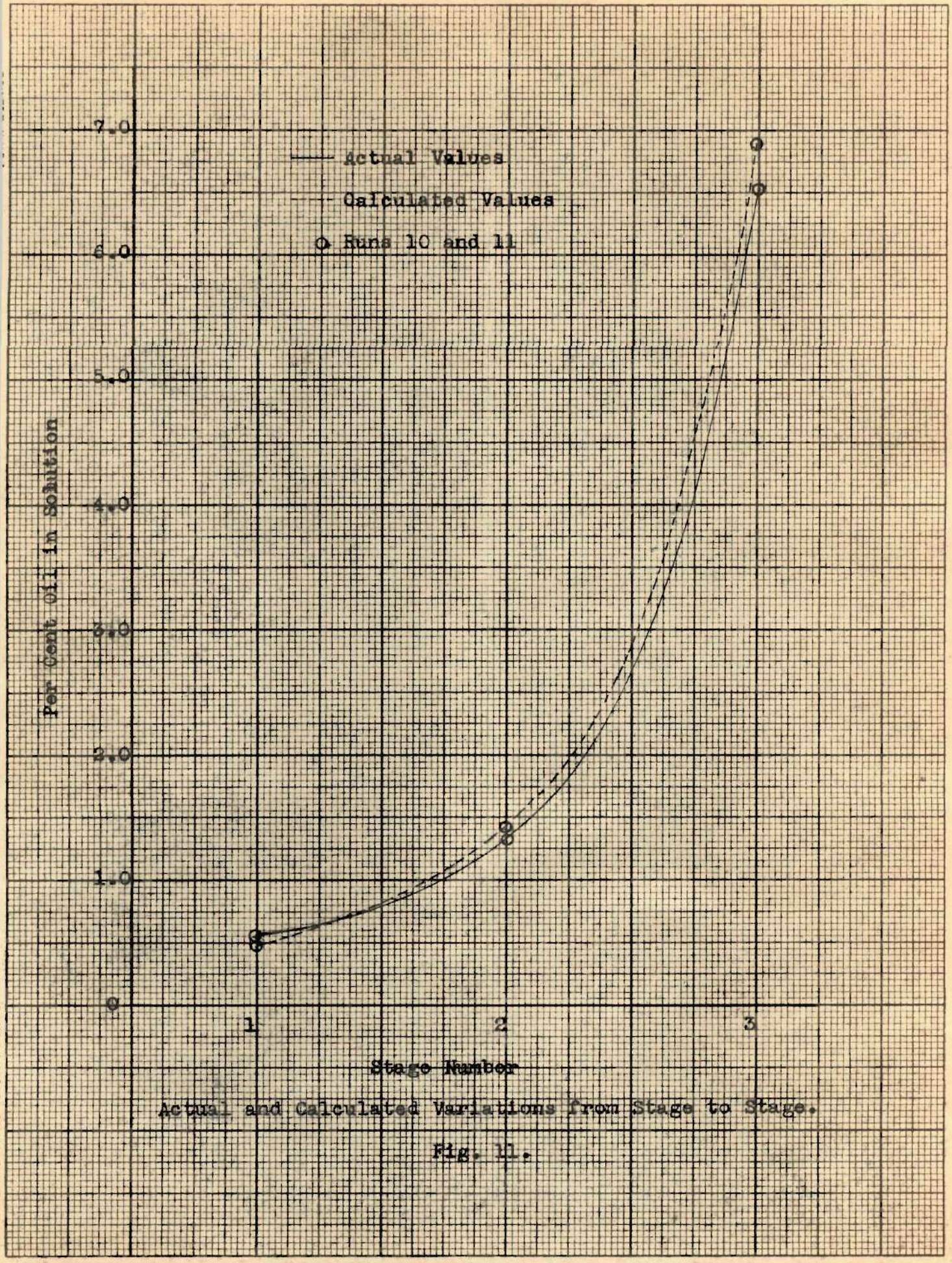
Fig. 9.

Figures 10 and 11 show the variation of actual from calculated concentrations within each unit for varying solid to solvent ratios. The actual data proved to be much more accurate at the lower ratios. This is easily understood from the fact that the disintegration of the cottonseed flakes had time to take place at the lower solvent rate, which was used in the first runs. Also, true countercurrent flow is approached only by use of a large number of stages. This effect was brought about by the lower ratios used in the last runs. As before, the approach to true equilibrium conditions is shown by the variation of the actual curves from the calculated curves.



Actual and Calculated Variations from Stage to Stage.

Fig. 10



Actual and Calculated Variations from Stage to Stage.

Fig. 11.

CONCLUSIONS AND RECOMMENDATIONS

From the results obtained, it is concluded that true multiple effect, continuous, countercurrent extraction is approached using the present apparatus with a low ratio of solvent to solid flow per unit time. By doubling the present number of extraction stages, the solid flow could be increased, and more accurate data obtained.

The point to point data, while varying up to thirty per cent from calculations in extreme cases, proved close enough to warrant further investigation. It is believed that with a larger number of extraction stages the point to point data would be much more accurate.

In further studies, the choice of a solid which holds its original size would be preferred. An excellent example of this is the soybean, which is being used considerably in commercial solvent extraction today.

Enough space has been left on each connecting line between the stages for installation of heater units for a study of temperature variation in extraction.

With these additions, the apparatus would provide a good means of investigating the effect of solvent rate of flow, solid rate of flow, temperature of solvent, and solid flake size upon the rate of extraction.

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APPENDIX

Table II

Comparison of Actual and Theoretical Values of Oil in Solution

Run No. 4

L_o/W_n: 7.60

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.30	.2
2	.59	.5
3	4.62	4.8

Run No. 5

L_o/W_n: 7.60

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.33	.2
2	.65	.5
3	4.45	4.8

Run No. 8

L_o/W_n: 6.49

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.49	.4
2	.96	.8
3	5.63	5.6

Run No. 9

L_o/W_n: 6.49

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.46	.4
2	.79	.8
3	5.40	5.6

Table II (Continued)

Run No. 10

L_O/W_n: 4.84

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.55	.5
2	1.31	1.4
3	6.53	6.9

Run No. 11

L_O/W_n: 4.84

Stage	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
1	.53	.5
2	1.31	1.4
3	6.78	6.9

Stage No. 1

L_O/W_n	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
7.60	.30	.2
7.60	.33	.2
6.49	.49	.4
6.49	.46	.4
4.84	.55	.5
4.84	.53	.5

Stage No. 2

L_O/W_n	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
7.60	.59	.5
7.60	.65	.5
6.49	.96	.8
6.49	.79	.8
4.84	1.31	1.4
4.84	1.31	1.4

Table II (Continued)

Stage No. 3

L_o/W_n	Weight % of Oil Remaining in Solution	
	Actual (Average)	Theoretical
7.60	4.62	4.8
7.60	4.45	4.8
6.49	5.63	5.6
6.49	5.40	5.6
4.84	6.53	6.9
4.84	6.78	6.9

Table III

Tabulation of Experimental Data

Run No. 4

Solvent Flow: 6100 Gm. per Hr.
Solid Flow : 800 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.50	-	-
8	-	.90	-
9	-	-	4.75
10	.30	-	-
11	-	.70	-
12	-	-	4.45
13	.30	-	-
14	-	.65	-
15	-	-	4.35
16	.30	-	-
17	-	.50	-
18	-	-	4.75
19	.30	-	-
20	-	.65	-
21	-	-	4.45
22	.30	-	-
23	-	.65	-
24	-	-	4.75
25	.30	-	-
26	-	.50	-
27	-	-	4.45

Run No. 5

Solvent Flow: 6100 Gm. per Hr.
Solid Flow : 800 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.35	-	-
8	-	.75	-
9	-	-	4.45
10	.30	-	-
11	-	.65	-
12	-	-	4.45

Table III (Continued)

Run No. 5 (Continued)

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
13	.30	-	-
14	-	.65	-
15	-	-	4.45
16	.35	-	-
17	.30	-	-

Run No. 8

Solvent Flow: 12,200 Gm. per Hr.
Solid Flow : 1,890 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.55	-	-
8	-	1.15	-
9	-	-	-
10	.60	-	-
11	-	1.05	-
12	-	-	5.60
13	.42	-	-
14	-	1.05	-
15	-	-	5.70
16	.41	-	-
17	-	.79	-
18	-	-	5.60

Run No. 9

Solvent Flow: 12,200 Gm. per Hr.
Solid Flow : 1,890 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.50	-	-
8	-	.75	-
9	-	-	5.60
10	.52	-	-
11	-	.91	-

Table III (Continued)

Run No. 9 (Continued)

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
12	-	-	5.35
13	.41	-	-
14	-	.75	-
15	-	-	5.12
16	.41	-	-
17	-	-	-
18	-	-	5.60

Run No. 10

Solvent Flow: 9100 Gm. per Hr.
Solid Flow : 1890 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.64	-	-
8	-	1.40	-
9	-	-	6.30
10	.55	-	-
11	-	1.32	-
12	-	-	6.61
13	.55	-	-
14	-	1.22	-
15	-	-	6.61
16	.51	-	-
17	-	1.31	-
18	-	-	6.61
19	.55	-	-

Run No. 11

Solvent Flow: 9100 Gm. per Hr.
Solid Flow : 1890 Gm. per Hr.

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
7	.78	-	-
8	-	1.22	-
9	-	-	6.61

Table III (Continued)

Run No. 11 (Continued)

Time (Min.)	Weight % Oil in Solution		
	Stage 1	Stage 2	Stage 3
10	.55	-	-
11	-	1.40	-
12	-	-	6.61
13	.55	-	-
14	-	1.22	-
15	-	-	6.90
16	.51	-	-
17	-	1.40	-
18	-	-	6.90
19	.53	-	-
20	-	-	-
21	-	-	6.90

Table IV

Sample Run

Run No. 4
Three Stage Extraction
Rate of Solvent Flow : 6,100 Gm./Hr.
Rate of Solid Flow : 800 Gm./Hr.

Time (Min.)	Stage	Refractometer	Weight % Oil
7	1	9.50	.50
8	2	9.54	.90
9	3	9.73	4.75
10	1	9.47	.30
11	2	9.53	.75
12	3	9.72	4.45
13	1	9.47	.30
14	2	9.52	.65
15	3	9.73	4.75
Remove Unit 1, Add Unit 4			
16	1	9.47	.30
17	2	9.50	.50
18	3	9.73	4.75
19	1	9.47	.30
20	2	9.50	.50
21	3	9.72	4.45
22	1	9.47	.30
23	2	9.52	.65
24	3	9.73	4.75
25	1	9.47	.30
26	2	9.50	.50
27	3	9.72	4.45
28	3	9.73	4.75
29	3	9.73	4.75

Analysis of P_1 by Soxhlet Extraction

	1	2
Time of Extraction	4 Hrs.	4 Hrs.
Solvent Used	Petroleum Ether	
Wt. Flakes, Gm.	.2246	.2197
Wt. Oil Removed	.0039	.0035
% Oil by Wt.	1.76	1.60

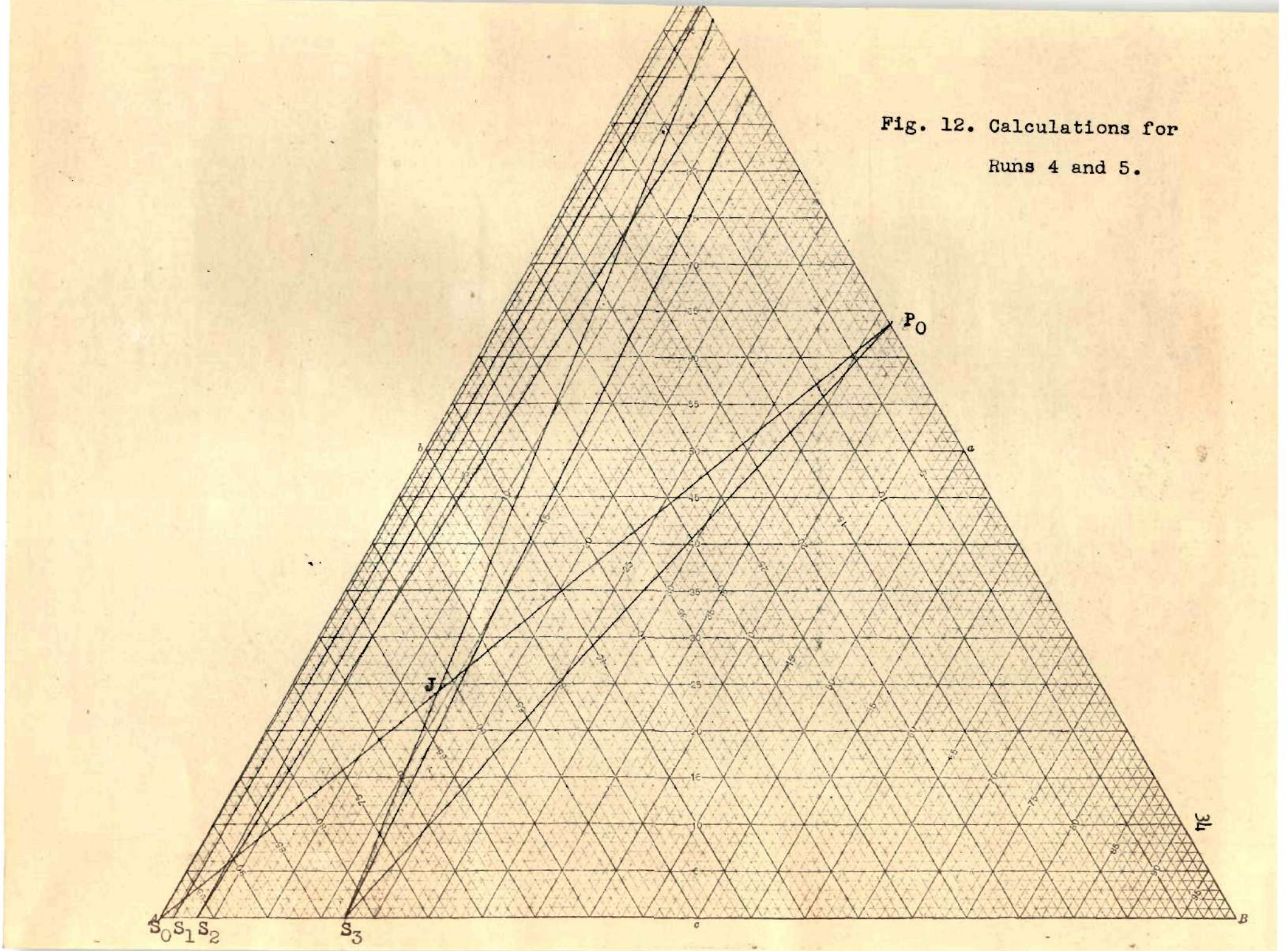


Fig. 12. Calculations for Runs 4 and 5.

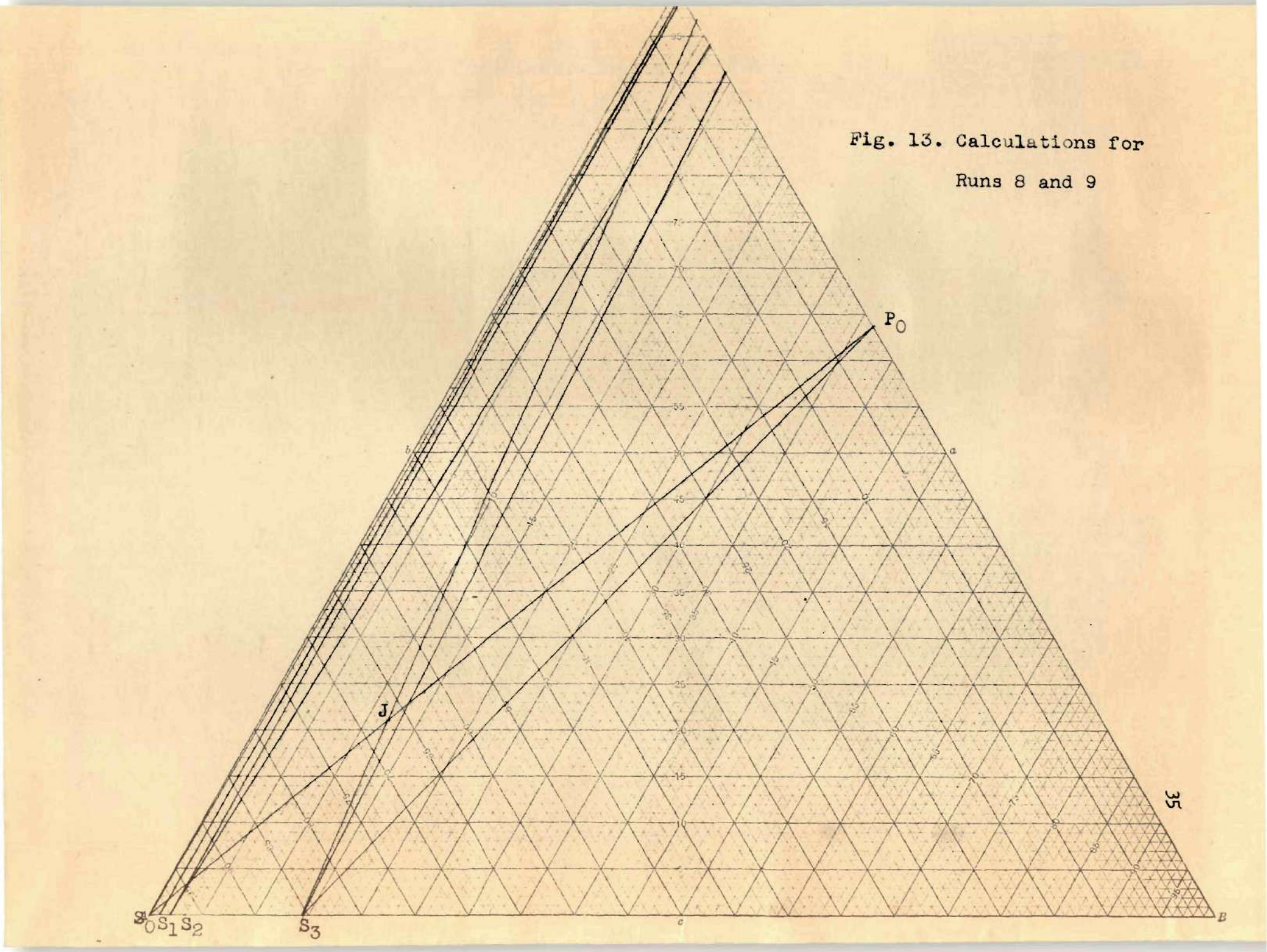


Fig. 13. Calculations for Runs 8 and 9

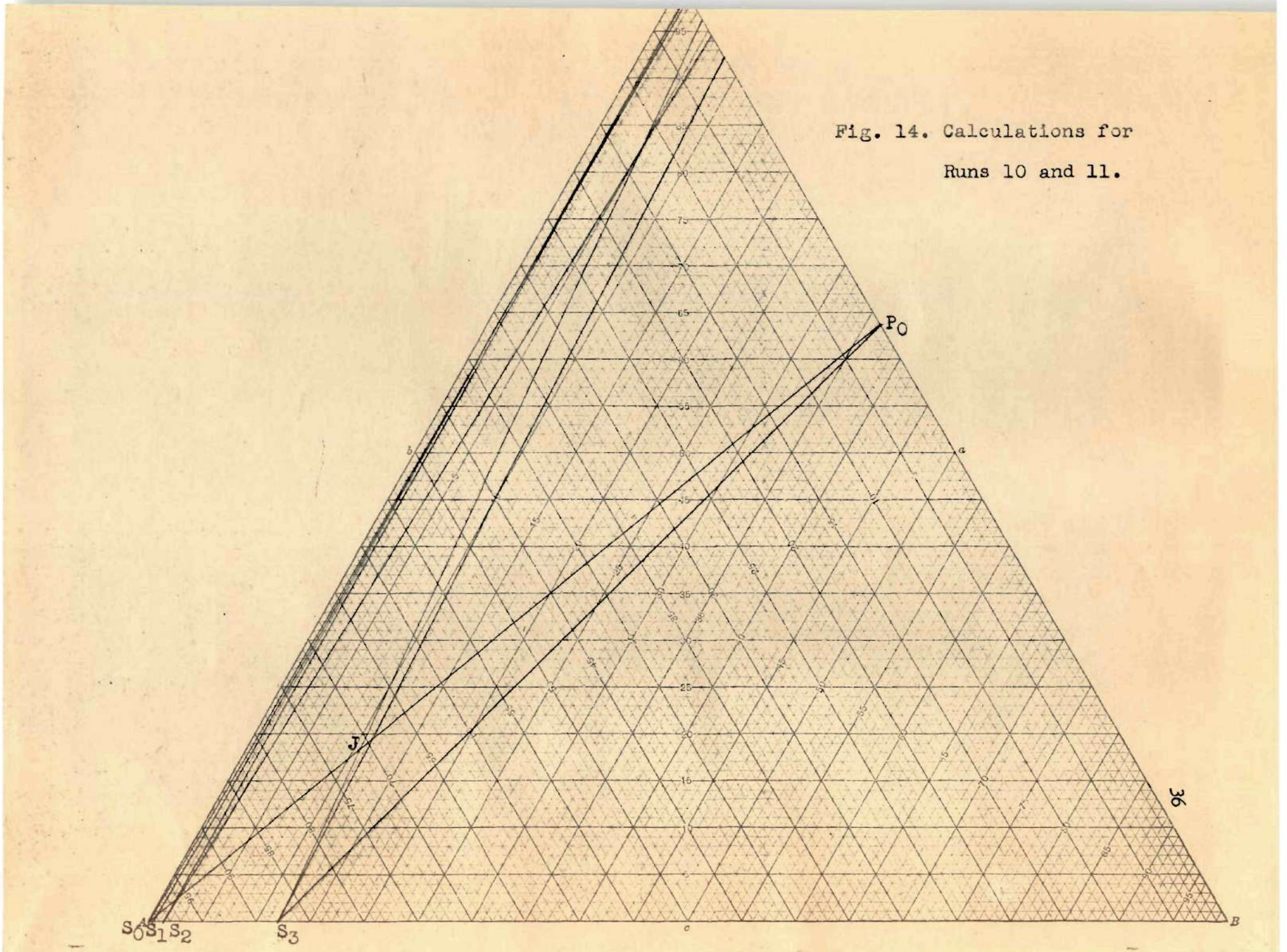
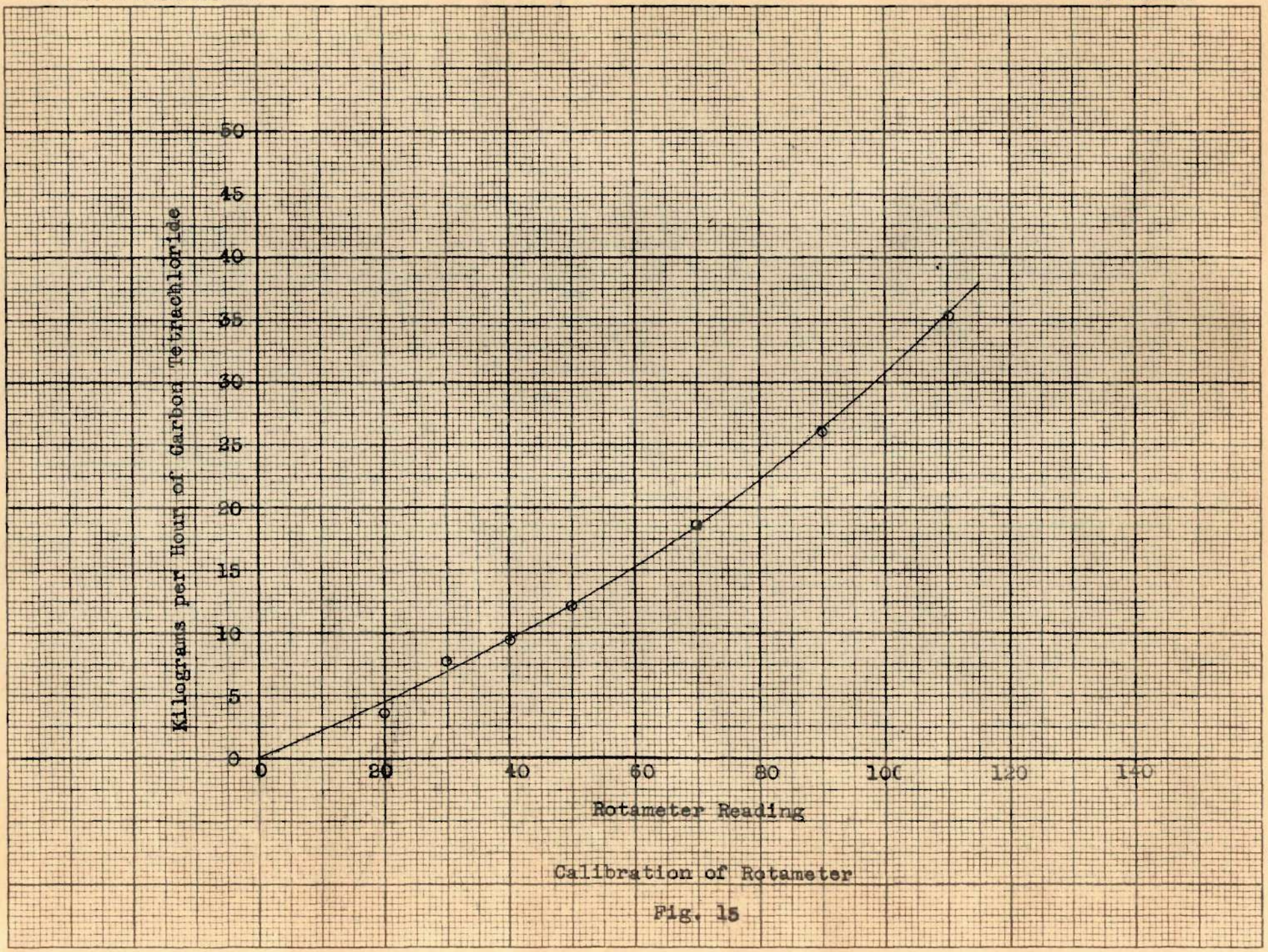


Fig. 14. Calculations for Runs 10 and 11.



Calibration of Rotameter

Fig. 15

Calibration of Refractometer

Fig. 16

Calibration of Refractometer

Fig. 17